TC 45

International Standard 289

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Rubber, unvulcanized — Determination of Mooney viscosity

Caoutchouc non vulcanisé — Détermination de la viscosité Mooney

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

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It cancels and replaces ISO Recommendation R 289-1963, of which it constitutes a technical revision.

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Rubber, unvulcanized — Determination of Mooney viscosity

1 Scope and field of application

This International Standard specifies a method of use of the shearing disk viscometer for measuring the Mooney viscosity of uncompounded or compounded rubbers.

2 References

ISO 471, *Rubber — Standard temperatures, humidities and times for the conditioning and testing of test pieces.*

ISO 1796, *Rubber, raw — Sample preparation.*

ISO 2393, *Rubber test mixes — Preparation, mixing and vulcanization — Equipment and procedures.*

3 Principle

The torque which has to be applied under specified conditions in order to rotate a metal disk in a cylindrical chamber formed from mating dies filled with rubber is measured. The resistance to this rotation, offered by the rubber, is expressed in arbitrary units as the Mooney viscosity of the test piece.

4 Apparatus

The essential parts of the apparatus (see figure 1) are:

a) two dies to form a cylindrical cavity;

b) a rotor;

c) a means for maintaining the dies at a constant temperature;

d) a means of maintaining a specified closure pressure;

e) a means for rotating the rotor at constant angular velocity;

f) a means for indicating the torque required to rotate the rotor.

The rotor and die cavity have the dimensions shown in table 1.

Table 1 — Dimensions of essential parts of the apparatus

Dimensions	Millimetres
Rotor diameter	38,10 ± 0,03
Rotor thickness	5,54 ± 0,03
Die cavity diameter	50,9 ± 0,1
Die cavity depth	10,59 ± 0,03

NOTE — Normally this rotor is called the large rotor.

It is permissible to use a smaller rotor where high viscosity makes this necessary. This small rotor should have the same dimensions as the large rotor except that the diameter should be 30,48 ± 0,03 mm. Results obtained with the small rotor are not identical with those obtained using the large rotor.

4.1 Dies

The two dies forming the cavity shall be formed from non-deforming unplated hardened steel of minimum Rockwell hardness HRC 60. The dimensions of the cavity are given in figure 1 and shall be measured from the highest surfaces.

For good heat transfer each die should preferably be made from only one piece of steel. The surfaces shall have radial V-grooves on the flat surfaces to prevent slipping. The grooves shall be spaced radially at 20° intervals and shall extend from an outer circle of diameter 47 mm to an inner circle of diameter 7 mm for the upper die and to within 1,5 mm of the hole in the lower die; each groove shall form a 90° angle in the die surface with the bisector of the angle perpendicular to the surface and shall be 1,0 ±0,1 mm wide at the surface (see figure 2).

Alternatively, each die may be formed from two pieces of steel with rectangular section grooves on the surfaces to prevent slipping. The grooves shall be 0,80 ± 0,02 mm wide, of uniform depth 0,30 ± 0,05 mm and spaced on 1,60 ± 0,04 mm centres. The flat surfaces of the dies have two sets of these grooves at right angles to each other (see figure 3).

NOTE — The two types of die may not give the same results.

4.2 Rotor

The rotor shall be fabricated from a non-deforming unplated hardened steel of minimum Rockwell hardness HRC 60. The rotor surfaces should be grooved as in the manner shown in figure 3. The rotor is fastened at right angles to a shaft having a diameter 10 ± 1 mm and of length such that in the closed die cavity the clearance above the rotor does not differ from that below by more than 0,25 mm. The rotor shaft shall bear on the spindle which turns the rotor shaft, not on the wall of the die cavity. The clearance at the point where the rotor enters the cavity should be small enough to prevent rubber leaving the cavity. A grommet, O-ring or other sealing device may be used as a seal at this point.

The eccentricity or runout of the rotor while turning in the viscometer shall not exceed 0,1 mm.

The relative speed of rotation between the rotor and dies shall be 0,209 ± 0,000 2 rad/s (2,00 ± 0,02 rev/min) unless otherwise stated.

4.3 Heating device

The dies are mounted on, or form part of, platens equipped with a heating device capable of maintaining the temperature of the platens and that of the dies to within ± 0,5 °C of the test temperature. After insertion of the sample the devices shall be capable of returning the temperature of the dies to within ± 0,5 °C of the test temperature within 5 min.

4.4 Temperature measurement

The test temperature is defined as the steady state temperature of the closed dies with the rotor in place and the cavity empty.

A temperature sensor shall be present in each die for measurement of die temperature. The sensor shall be located for the best possible heat contact with the dies, i.e. heat gaps and other heat resistance shall be excluded. The axes of the sensors shall be at a distance of 3 to 5 mm from the working surface of the dies and 15 to 20 mm from the rotation axis of the rotor. The temperature measuring system shall be capable of indicating temperature to an accuracy of 0,25 °C.

4.5 Die closure

The dies may be closed and held closed by hydraulic, pneumatic or mechanical means. A force of 11,5 ± 0,5 kN shall be maintained on the dies during the test.

A greater force may be required to close the dies when rubbers of high viscosity are tested; at least 10 s before starting the viscometer the force shall be reduced to 11,5 ± 0,5 kN and maintained at this level throughout the test.

NOTE — A closing and holding force of 8,0 kN is allowable if the rubber has a viscosity less than 50 Mooney units.

For all types of closing devices, a piece of soft tissue paper not thicker than 0,04 mm placed between the mating surfaces should show a continuous pattern of uniform intensity when the dies are closed. A non-uniform pattern indicates incorrect adjustment of die closure, worn or faulty mating surfaces or distortion of dies; any of these conditions result in leakage and erroneous results.

4.6 Torque measurement and calibration

The torque required to turn the rotor is recorded or indicated on a linear scale graduated in Mooney units. The reading shall be zero when the machine is run empty and 100 ± 0,5 when a torque of 8,30 ± 0,02 N·m is applied to the rotor shaft. Therefore a torque of 0,083 N·m is equivalent to 1 Mooney unit. The scale shall be capable of being read to 0,5 Mooney units. Variation from zero shall be less than ± 0,5 Mooney units when the machine is running with the rotor in place, and the dies closed and empty.

The shearing disk viscometer shall be calibrated while the machine is running at the test temperature. A suitable method for the calibration of most machines is as follows.

The scale is calibrated by applying certified masses fastened with flexible wire of diameter 0,45 mm to an appropriate rotor. During calibration the rotor is turned at 0,209 rad/s and the platens are at the specified test temperature.

NOTES

1 In order to check linearity, intermediate masses may be used to give scale readings of 25, 50 and 75 Mooney units, respectively.

2 A sample of butyl rubber of certified Mooney viscosity can be used to check whether the machine is working correctly. Measurement may be carried out at 100 or 125 °C for 8 min. The rubber should be obtained from a national testing office or from the National Bureau of Standards, Washington, DC, USA (designation IIR-NBS-388).

5 Preparation of test pieces

For uncompounded rubbers, the test portion should be prepared according to ISO 1796 (see the annex of ISO 289). For compounded rubbers which are to be tested for reference purposes, the test portion shall be taken from a compound prepared according to ISO 2393 and the material standard relevant to the rubber.

The test portion shall be allowed to rest at standard laboratory temperature (see ISO 471) for at least 30 min before testing is carried out. Testing shall be commenced not later than 24 h after homogenization.

NOTE — The Mooney viscosity is affected by the manner in which the rubber is prepared and the conditions of storage prior to test. Accordingly, the prescribed procedure in methods for evaluating the particular rubber should be followed rigorously.

The test piece shall consist of two disks of rubber, of diameter about 50 mm and of thickness approximately 6 mm, sufficient to fill completely the die cavity of the viscometer. The rubber disks shall be as free as possible from air and from pockets that may trap air against the rotor and die surfaces. A hole is pierced or cut through the centre of one disk to permit the insertion of the rotor stem.

6 Temperature and duration of test

The test shall be made at 100 ± 0,5 °C or 125 ± 0,5 °C for 4 min or 8 min as indicated in table 2 (see ISO 1796).

Table 2 — Test conditions for different types of rubber

Type of rubber	Test temperature	Running time of rotor
	°C	min
NR	100	4
IIR, BIIR, CIIR	100 or 125*	8
EPDM, EPM	125	4
Other synthetic rubbers, black masterbatch, compounded rubber and reclaimed material	100	4

* A temperature of 125 °C should be used whenever the specimen has a viscosity higher than 60 ML (1 + 8) 100 °C.

7 Procedure

Heat the dies and rotor to the test temperature and allow them to reach a steady state. Open the dies, insert the rotor stem through the hole in the test piece and place the rotor in the viscometer. Place the second test piece centrally on the rotor and close the dies as quickly as possible.

NOTE — A heat stable film, for example of polyester, of thickness approximately 0,03 mm may be inserted between the rubber and die surfaces to facilitate removal after test of low viscosity or sticky materials. The use of such film may affect the test results.

Note the time at which the dies are closed and allow the rubber to preheat for 1 min. Start the rotor; the running time shall be as indicated in table 2. If the viscosity is not recorded continuously observe the scale during the 30 s interval preceding the specified reading time and report the minimum value, to the nearest 0,5 unit, as the viscosity. For reference purposes, take readings at 5 s intervals from 1 min before to 1 min after the specified time. Draw a smooth curve through the minimum points of the periodic fluctuations or through all the points if there are no fluctuations. Take the viscosity as the point where the curve intersects the time specified. If a recorder is used, take the viscosity from the curve in the same manner as specified for the plotted curve.

NOTE — To check if the temperature of the test piece is at the test temperature at the preferred test time (4 min or 8 min) two thermocouple measurement probes may be inserted into the sample as shown in figure 4. In a preliminary test with the test piece to be measured, the rotor is stopped after a running time of 3,5 or 7,5 min, respectively, and immediately after the resulting standstill the two measurement probes are inserted so that after 4 or 8 min the two mean test piece temperatures can be read off. The temperature tolerance should be between 0,0 and −2 °C.

The temperature gradients in the test piece and rate of heat transfer vary among viscometers, particularly if different types of heating are employed. Therefore, the values obtained with different viscometers may be expected to be more comparable after the rubber has attained the test temperature. Usually this condition is reached within 10 min after the die cavity is closed.

8 Expression of results

8.1 Format

Results of a typical test should be reported in the following format:

50 ML (1 + 4) 100 °C

where

50 M is the viscosity, in Mooney units;

L indicates that the large rotor was used (S would indicate use of the small rotor);

1 is the preheating time, in minutes, before starting the rotor;

4 is the running time, in minutes, after starting the rotor at which the final reading is taken;

100 °C is the temperature of the test.

8.2 Precision

8.2.1 Repeatability

For the range of viscosity from 40 to 60 Mooney units, the standard deviation of repeatability (same operator, same apparatus, same laboratory and short intervals of time) of a uniform sample of rubber is about 0,2 Mooney units. Variation in sample preparation results in standard deviations of about 1 Mooney unit.

The standard deviation of repeatability increases with increasing viscosity.

The use of test pieces of constant volume decreases the standard deviation of repeatability.

8.2.2 Reproducibility

For the range of viscosity from 40 to 60 Mooney units, the standard deviation of reproducibility (different operators, different apparatus of the same design, different laboratories and/or different times) of a uniform sample of rubber is up to 2 Mooney units.

In special cases variability among laboratories may cause even larger variations in results. Part of the interlaboratory variability is due to sample preparation, and part to differences in calibrating or adjusting the viscometer.

The standard deviation of reproducibility increases with increasing viscosity.

The use of test pieces of constant volume decreases the standard deviation of reproducibility.

9 Test report

The test report shall include the following information:

a) a full description and identification of the sample tested, including

1) its origin,

2) preparation of test pieces, for example, procedure of milling (see ISO 1796 or the annex of ISO 289),

3) details of compounded rubbers, if applicable;

b) a reference to this International Standard;

c) a description of the apparatus used, including

1) model and manufacturer of equipment,

2) rotor size,

3) type of dies;

d) details of the conditions of test (see clause 8), including

1) temperature of test (100 or 125 °C),

2) preheat time, if other than 1 min,

3) running time (4 min or 8 min),

4) rotor speed, if other than 0,209 rad/s,

5) closing force, if other than 11,5 kN;

e) the value of the Mooney viscosity (see clause 8);

f) any operation not included in this International Standard, or regarded as optional;

g) the date of the test.

Dimensions in millimetres

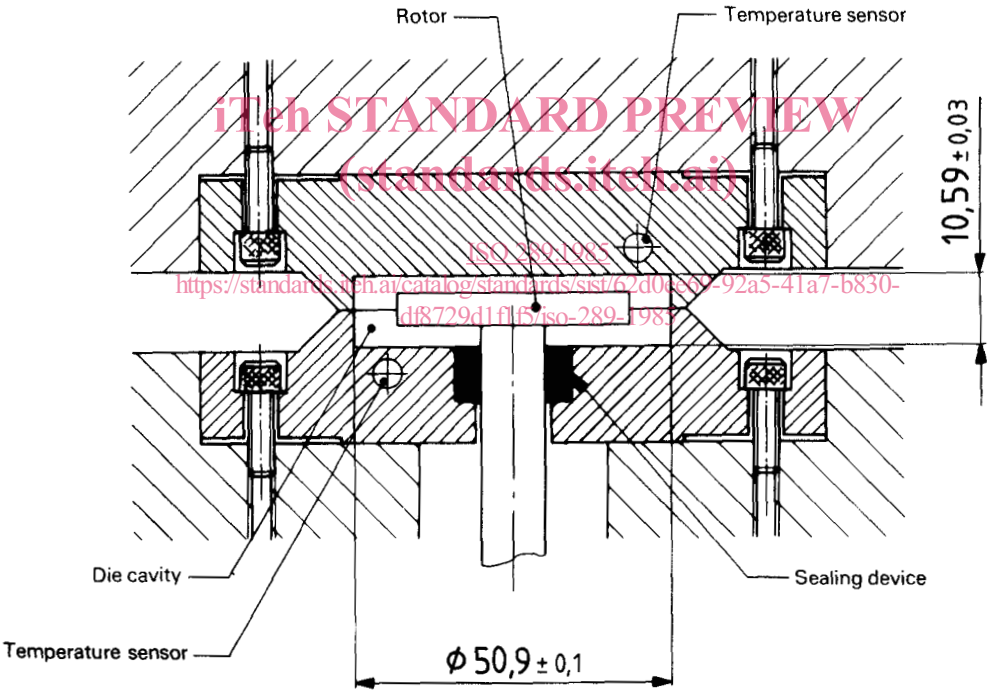


Figure 1 — Typical shearing disk viscometer

Dimensions in millimetres

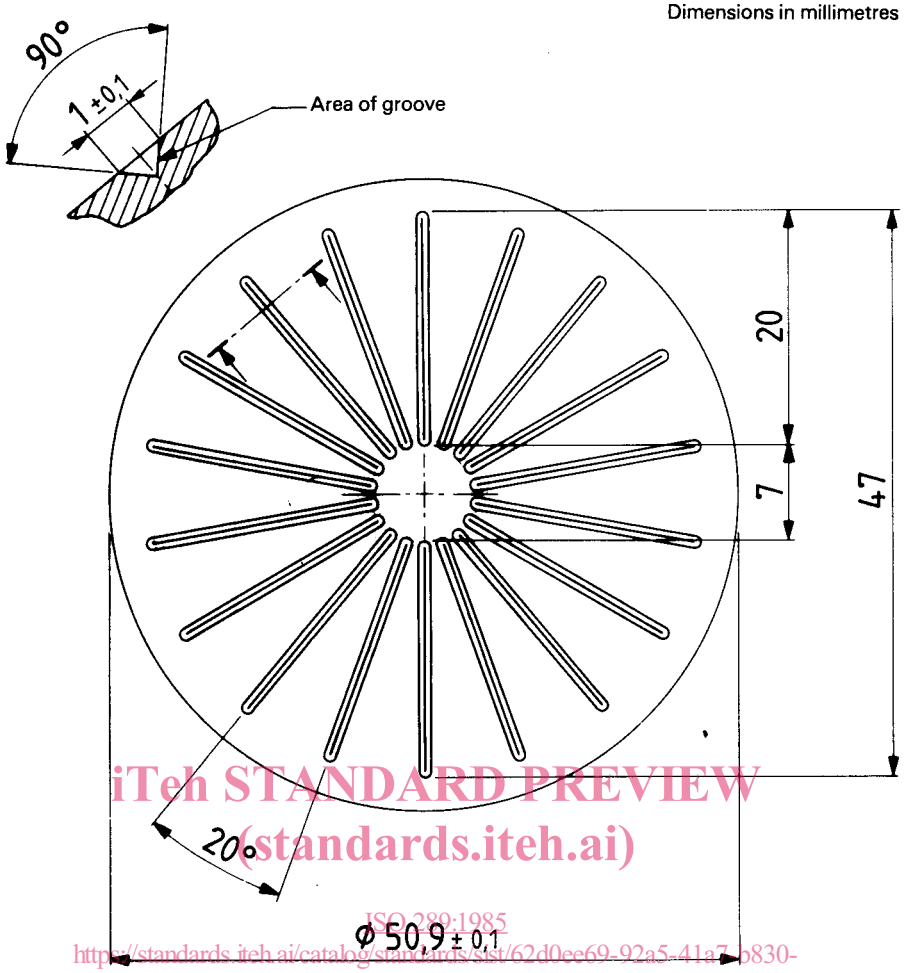


Figure 2 — Die with radial V-grooves

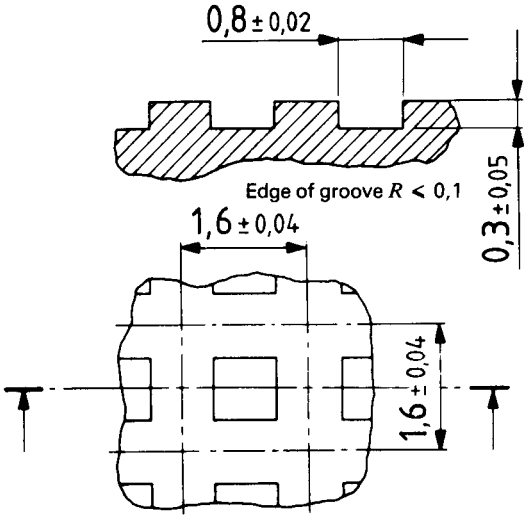
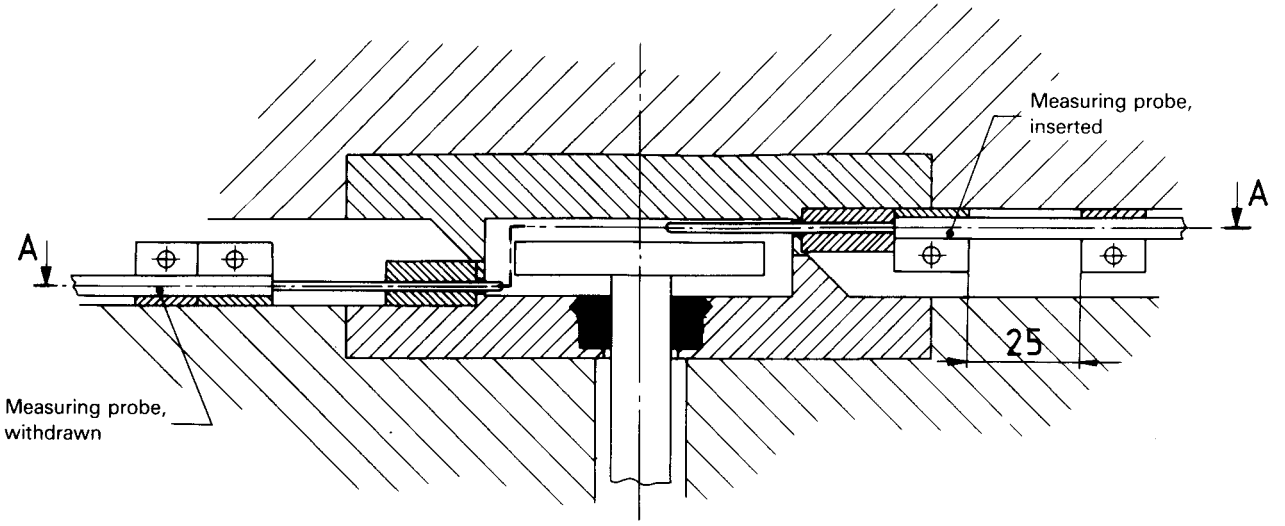


Figure 3 — Grooves with rectangular section of die and rotor

Dimensions in millimetres



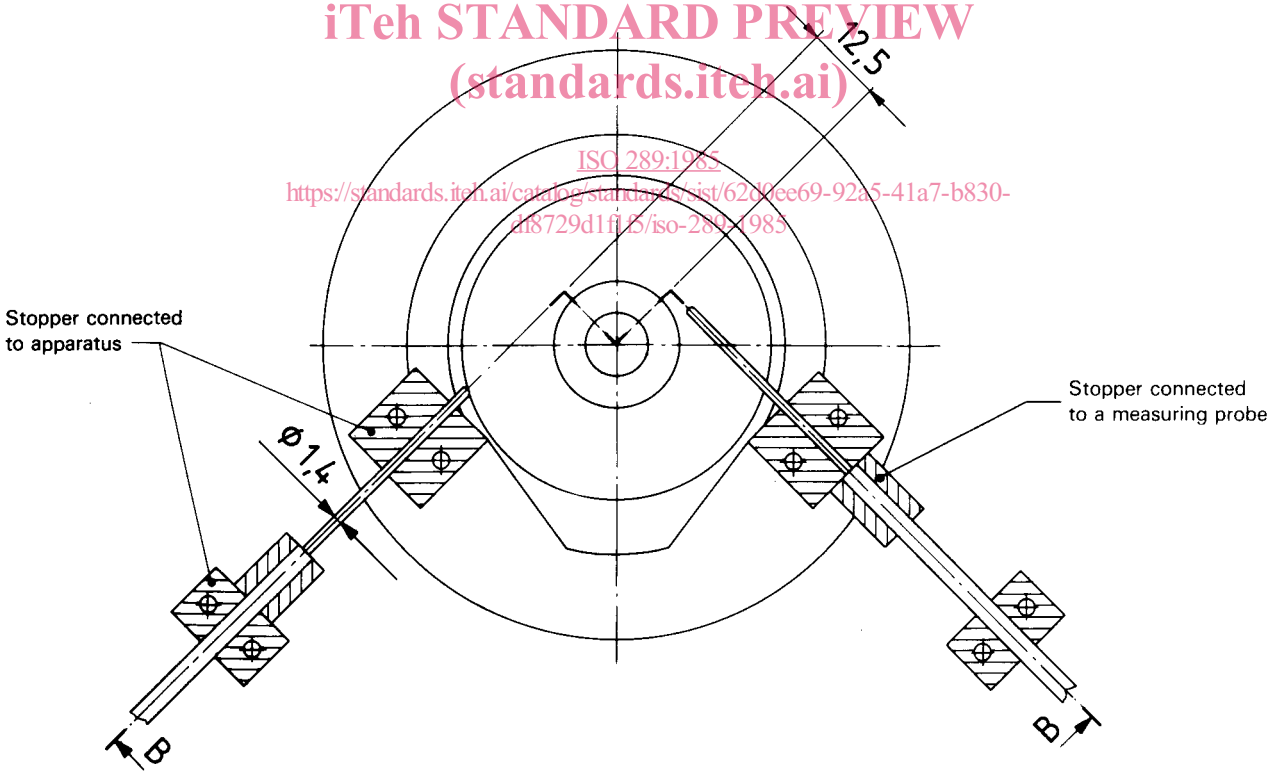


Figure 4 — Measuring probe design

Annex

Methods of sample preparation for Mooney viscosity testing

(Forms an integral part of the Standard.)

The methods of sample preparation for Mooney viscosity testing, according to ISO 1796, are summarized in table 3.

Table 3 — Sample preparation for different types of rubber

Type of rubber	Mill roll surface temperature	Nip width	Number of passes
	°C	mm	
NR	70 ± 5	1,3 ± 0,15	10
Synthetic rubber, except IIR, BIIR and CIIR (see below), and the following	50 ± 5	1,4 ± 0,15	10
BR	35 ± 5	1,4 ± 0,15	10
CR	20 ± 5	0,4 ± 0,05	2
EPDM, EPM	35 ± 5	1,4 ± 0,15	10
IIR, BIIR and CIIR [1]	— [2]		
Black masterbatch	50 ± 5	1,4 ± 0,15	10
Compounded rubber and reclaimed material	— [2]		

1) When butyl rubbers (IIR, BIIR and CIIR) are tested in crumb form they must be massed by the method given for synthetic rubbers.

2) No milling.

NOTE — The alternative mill roll surface temperatures for BR, CR, EPDM and EPM are given in notes in ISO 1796 and are therefore optional.